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IS 7212 (1974): Methods of determination of copper [CHD 1: Inorganic Chemicals]



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Indian Standard

(Reaffirmed 1981)

METHODS OF DETERMINATION OF COPPER

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**BUREAU OF INDIAN STANDARDS
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*Indian Standard***METHODS OF DETERMINATION OF COPPER**

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Indian Standard

METHODS OF DETERMINATION OF COPPER

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 7 February 1974, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 Copper in micro and macro quantities is required to be determined in various products. Numerous methods are available for its determination in all ranges but a few have been found to be more convenient and suitable and these are now used in preference to others. For example, electro-deposition on a platinum gauze cathode is an excellent method but is time consuming. This is used for determining copper in macro quantities where large batch of samples can be simultaneously electrolyzed in a battery of electrolytic cells. It is also used where the volumetric iodide method cannot be employed due to any reason. The volumetric iodide method is a very rapid and convenient method for determining macro quantities of copper. For micro-analysis of copper, several organic reagents are used but more common reagent is the diethyldithiocarbamate. Biquinoline [2, 2 biquinolyl or 2-(2-quinolyl)-quinoline] is also considered to be most suitable but its use is under investigation with the Committee.

0.3 This standard is intended to achieve uniformity of the method for the determination of copper prescribed in various Indian Standards.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS:2-1960*.

1. SCOPE

1.1 This standard prescribes electrolytic, volumetric and colorimetric methods for the determination of copper in micro and macro quantities.

*Rules for rounding off numerical values (*revised*).

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (see IS: 1070-1960*) or demineralized water (demineralized through ion-exchange resin) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. PREPARATION OF SOLUTION

3.1 Before carrying out the analytical procedures, the copper in test samples has to be brought into solution. In most cases, attack by nitric acid is enough; occasionally sulphuric acid, fuming, has to follow this attack. In case of organic matter and biological samples attack by sulphuric acid and an oxidizing agent (usually hydrogen peroxide) is required but direct ashing by ignition can also be done. Highly siliceous samples may be opened up with hydrofluoric acid and sulphuric acid treatment or by fusion with pyrosulphate. In all cases, the whole of the copper comes into the acidic solution and insoluble residue, if any, can be filtered out. There is no volatile loss of copper in ashing, fusion or during fuming of sulphuric acid. The ashing is done at a temperature not exceeding 600°C.

4. ELECTROLYTIC DETERMINATION OF COPPER

4.0 Outline of the Method — Electrolysis is carried out in a weak acidic solution and copper deposited on a platinum gauze electrode is weighed.

4.1 Apparatus

4.1.1 Electrolytic Apparatus — A suitable apparatus for electrolysis with or without agitation of the electrolyte.

4.1.2 Electrodes — Platinum gauze electrode having a surface area of about 1 cm².

4.2 Reagents

4.2.1 Solvent-Acid Mixture — Add 200 ml of concentrated sulphuric acid (see IS: 266-1961†) to 500 ml of water, then add 200 ml of concentrated nitric acid (see IS: 264-1968‡).

4.2.2 Dilute Nitric Acid — 1:1 by volume.

*Specification for water, distilled quality (revised).

†Specification for sulphuric acid (revised).

‡Specification for nitric acid (first revision).

4.3 Procedure

4.3.1 Weigh accurately the material containing between 0.1 to 0.3 g of copper and transfer to a beaker provided with a watch glass. Dissolve the material in the solvent-acid mixture. Generally 20 ml of the mixture is required per gram of the sample. Add further 10 ml of the mixture. When the dissolution is complete, boil vigorously by adding a pinch of urea or sulphamic acid to drive off the nitrous fumes and cool. Wash the watch glass and sides of the beaker carefully. Dilute the contents to about 200 ml with water.

4.3.2 Clean the electrodes by immersing in dilute nitric acid, followed by washings with water, immersing in absolute alcohol and finally drying at 105°C for about 10 minutes. Weigh the cathode accurately and fit the electrodes in the electrolysis apparatus so that they are as near to the bottom of the beaker as possible. Cover the beaker with a pair of split watch glass and electrolyze for 18 hours at 1.0 ampere with a static electrolyte or for 2 to 3 hours at 4 amperes with agitation of the solution. At the end of this period wash the cover glass and sides of the beaker and continue the electrolysis for another 30 minutes. This will increase the volume of the solution, thus immersing a little more portion of the cathode surface, and if no more copper is deposited on this freshly immersed surface, remove the cover glass and lower the beaker with the current still switched on, simultaneously washing the electrodes with a jet of water from a wash bottle. Switch off the current and remove the cathode, immerse it in absolute alcohol and dry at 105°C for 10 minutes. Weigh the cathode and calculate the increase in mass due to metallic copper.

4.4 Calculation

$$\text{Copper (as Cu), percent by mass} = \frac{M_2 - M_1}{M} \times 100$$

where

M_2 = mass in g of the cathode after electro-deposition,

M_1 = mass in g of the cathode before electro-deposition, and

M = mass in g of the material taken for the test.

NOTE 1—All the copper should be removed from the solution but, as an additional safeguard, examine the electrolyte as follows:

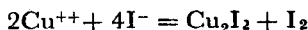
Add 20 ml of citric acid solution (30 percent, m/v) followed by ammonium hydroxide (18 N) until the solution is distinctly alkaline, then add 10 ml in excess, followed by 10 ml of sodium diethyldithiocarbamate (0.1 percent, m/v). If a brown colour is produced, extract with carbon tetrachloride, determine the copper approximately by colorimetric method by visual comparison (see 6) and add it to the above results.

NOTE 2—Any insoluble residue, after the initial acid treatment, should be filtered, ignited, fused with potassium pyrosulphate, the melt dissolved in water and added to the electrolyte before electrolysis. If lead is known to be present, it may be precipitated as sulphate in the solution of the sample, filtered, washed well with sulphuric acid (2 percent) and rejected.

5. VOLUMETRIC IODIDE METHOD

5.0 Outline of the Method— This is a rapid method of high degree of accuracy but it is subject to interference by many other elements. Most of these interferences lead to very slight error only if proper pH is maintained at the time of titration.

Cupric ions react with iodide ions in which cuprous iodide and equivalent amount of iodine are formed as per the reaction



The liberated iodine is determined by titration against standard thio-sulphate solution. The reaction and the titration are best carried out at a pH of 3.5 to 4.0. At pH widely different from this, the results obtained are not accurate.

5.1 Reagents

5.1.1 Ammonium Hydroxide Solution — 18 N.

5.1.2 Ammonium Bifluoride Solution — nearly saturated. Keep in polyethylene containers.

5.1.3 Potassium Iodide Solution — 50 percent (m/v), freshly prepared solution.

5.1.4 Standard Sodium Thiosulphate Solution — 0.1 N (see 55 of IS: 2316-1968*).

5.1.5 Starch Solution — fresh, 0.5 percent (m/v) (see SI No. 4 of Table VII of IS: 2263-1962†).

5.1.6 Copper Metal — electrolytically pure.

5.1.7 Urea — solid.

5.1.8 Potassium Thiocyanate — solid.

5.2 Procedure

5.2.1 Make the solution of the material in nitric acid. If the material is insoluble in nitric acid, sulphuric acid may be also added. But, if hydrochloric acid is used, this reagent should be removed by evaporation with sulphuric acid before neutralization. Transfer a known aliquot of solution containing up to 0.3 g of copper in a 250-ml stoppered conical flask. Remove the stopper, evaporate the solution to about 2 or 3 ml carefully, taking care that no part of the solution is evaporated to dryness. Dilute to about 50 ml. Add 1 g of urea and boil

*Methods of preparation of standard solutions for colorimetric and volumetric analysis (first revision).

†Methods of preparation of indicator solution for volumetric analysis.

for about a minute with stopper on. Cool, add ammonium hydroxide drop by drop with swirling, until a permanent precipitate is formed and a faint smell of ammonia is noticed in the solution after keeping it for a minute or two. (This precipitate is usually, though not always, brown due to ferric hydrated oxide; but the supernatant liquid is always, more or less, bluish or blue.) Avoid adding too much excess of ammonia. Add ammonium bifluoride solution drop by drop with thorough mixing until the brown precipitate redissolves and any yellow or red tinge in the solution disappears. Add 1 ml more.

Add about 4 to 6 ml of potassium iodide solution and mix with stopper on. Keep for 2-3 minutes (preferably in dark). Titrate the liberated iodine with standard thiosulphate solution until the iodine colour lightens, then add about 5 ml of starch solution and 2-3 g of potassium thiocyanate and continue the titration until the starch colour is discharged. Add about 2 ml of potassium iodide solution more and mix. If the starch colour reappears, continue the titration as above. Repeat until on addition of more potassium iodide; starch colour does not reappear.

NOTE 1 — Starch solution contains two kinds of starch, one of which is present in major proportion and gives blue colour with iodine. The other type is present in minor proportion only and gives a red colour with iodine. The red colour is more stable than blue. On titration, the original colour, which is almost blue, gradually changes to violet and then to red as the less stable blue starch colour is progressively discharged by thiosulphate. At the end point, it is the red starch colour which disappears.

NOTE 2 — The addition of solid urea and boiling for a minute and cooling can be omitted if the sample solution is known to be free from nitric acid.

5.2.1.1 Calculation

$$\text{Copper (as Cu), percent by mass} = \frac{6.354 \times V \times N}{M}$$

where

V = volume in ml of standard sodium thiosulphate solution used in titration,

N = normality of standard sodium thiosulphate solution, and

M = mass in g of the material taken for the test.

6. PHOTOMETRIC (COLORIMETRIC) METHOD

6.0 Outline of the Method—Sodium diethyldithiocarbamate by reaction in acid, neutral or alkaline solution, gives the copper salt by diethyldithiocarbamic acid which has a golden yellow colour. The complex is extracted with carbon tetrachloride and photometrically determined. By this treatment all interfering elements except bismuth are eliminated.

6.1 Apparatus

6.1.1 Photometer — Any suitable photoelectric colorimeter.

6.2 Reagents

6.2.1 Ammonium Citrate Solution — 40 percent (m/v).

6.2.2 Sodium Diethyldithiocarbamate Solution — 0.1 percent (m/v).

6.2.3 Standard Copper Solution — Clean the surface of a copper wire with sand paper. Dissolve 1 000 g of pure copper in dilute nitric acid. Heat the solution nearly to dryness to drive off the acid. Add about 10 ml of water and heat again nearly to dryness. Take up the residue in water, add 25 ml of 1 N sulphuric acid and dilute to 1 000 ml. One millilitre of this solution contains 0.1 mg of copper (as Cu).

Alternatively, dissolve 3.928 g of cupric sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in sufficient water containing 1 or 2 ml of concentrated sulphuric acid and dilute to 1 000 ml. One millilitre of the solution contains 1.0 mg of copper (as Cu).

6.2.4 Ammonium Hydroxide — 18 N.

6.2.5 Carbon Tetrachloride

6.3 Procedure

6.3.1 Prepare the solution of the sample as prescribed in the relevant material specification. Transfer an aliquot containing not more than 0.05 mg of copper to a 100-ml separating funnel. Add 2 ml of ammonium citrate solution and adjust the pH to 8.5 with ammonium hydroxide. The presence of ammonium ions helps to deionize iron, if present, and to prevent rapid fading of developed colour. Add 10 ml of sodium diethyldithiocarbamate solution and develop the colour. Extract the coloured complex with 5 to 10 ml of carbon tetrachloride. Separate the carbon tetrachloride layer from aqueous layer and centrifuge for 5 minutes to separate the water droplets. Transfer the carbon tetrachloride solution to a photometric cell and measure its absorbance at the wavelength of maximum absorption, 440 nm relative to reagent blank. Calculate the corrected absorbance by subtracting the reading obtained for the solution containing no copper.

6.3.1.1 Transfer to a series of 100-ml separating funnels aliquots of standard copper solution corresponding to 0, 5, 10, 15, 20, 25 g of copper and proceed exactly as prescribed in **6.3.1**. Plot a graph of corrected absorbance of solutions against their copper contents.

NOTE — If bismuth is suspected, it has to be separated or otherwise accounted for.

6.3.1.2 Calculation— Calculate the corrected absorbance by subtracting the value obtained for the blank from that obtained for the test solution and read from the calibration curve the corresponding mass of copper.

$$\text{Copper (as Cu), percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of copper as determined in the given aliquot of the test solution, and

M_2 = mass in g of the material present in the aliquot of the test solution.

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AMENDMENT NO. 1 JULY 1979

TO

IS : 7212-1974 METHODS OF DETERMINATION OF COPPER

[For micro-analysis of copper, the reagent prescribed is diethyldithiocarbamate. At the time of preparation of this standard, the method employing biquinoline was under investigation. The Committee, responsible for the preparation of the standard, decided to incorporate this method in the standard through this amendment.]

Alteration

(Page 2, clause 0.2, last sentence) — Substitute the following for the existing sentence:

‘Diethyldithiocarbamate method has possibility of interference by bismuth, cobalt and lead and in such cases biquinoline method shall be employed. In case of dispute, the biquinoline method shall be the referee method.’

Addendum

(Page 8, clause 6.3.1.2) — Add the following new matter after 6.3.1.2:

‘ 7. BIQUINOLINE METHOD

7.0 Outline of the Method — Copper reduced to cuprous state by hydroxylamine hydrochloride, reacts with 2, 2'-biquinoline solution in normal or isoamyl alcohol, to give a pinkish-violet colour in the organic liquid layer. This colour is stable and may be measured visually or spectrophotometrically over a wide range and is proportional to the amount of copper present. Best working pH range is between 5 to 6. Quantitative results can, however, be obtained if the copper content is limited to 50 μg . No other metal interferes.

NOTE — This method is suitable for materials of low copper content and will need some modification depending on nature of material. Copper in iron ore, steel or special alloy steel cannot be determined unless iron is reduced with excess of sulphurous acid. Hydroxylamine hydrochloride will be inadequate for the purpose. Organic matter, if present should be burnt.

7.1 Apparatus

7.1.1 Nessler Cylinders — See IS : 4161-1967*.

*Specification for Nessler cylinders.

7.1.2 Spectrophotometer — Visible range 400 to 800 nm.

7.2 Reagents

7.2.1 Buffer Solution — Dissolve 400 g of sodium acetate trihydrate, 20 g of hydroxylamine hydrochloride and 100 g of sodium tartrate in one litre of water. The pH of the buffer should be adjusted between 6 to 7. The buffer should be cleaned by shaking with 10 ml of biquinoline solution in a separating funnel of one litre capacity. If the organic liquid becomes pinkish violet it should be removed and the process repeated till the organic liquid layer becomes perfectly colourless.

7.2.2 Biquinoline Solution — Dissolve 0.2 g of 2, 2'-biquinoline reagent in 800 to 900 ml of isoamyl alcohol by warming. Cool and make up to 1 litre with the same alcohol. Normal amyl alcohol can also be used in place of isoamyl alcohol.

NOTE — No special storage precautions are necessary for the above reagents but it may be useful to keep them away from strong sunlight. The buffer may be kept in a refrigerator and biquinoline solution in amber coloured tightly stoppered bottle.

7.2.3 Standard Copper Solution

- a) *100 micrograms per ml* — Dissolve 0.200 g of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in 0.1 N hydrochloric acid and dilute to 500 ml with the acid.
- b) *10 microgram per ml* — Transfer 10 ml of standard solution (a) to a 100 ml volumetric flask and dilute to 100 ml with 0.1 N hydrochloric acid.

7.3 Procedure

7.3.1 Visual — Transfer to a number of Nessler cylinders aliquots of standard copper solution [7.2.3(b)] corresponding to 0.0, 0.2, 0.5, 1.0, 2.0, 3.0, 4.0, and 5 μg of copper. Add 10 ml of buffer solution and mix well. Then add 2 ml of biquinoline solution, tightly stopper and shake vigorously for 2 minutes, allow the layers to separate. For semiquantitative analysis the colour obtained in the organic liquid layer, in case of test sample similarly treated, is visually compared against similar colour in case of standards and the value of the standard to which it most nearly matches, is taken to be the value for the test sample. The test sample will pass the limit test if the colour in the organic liquid layer, in case of test sample, is equal to or less than that in case of standard solution.

7.3.2 Spectrophotometrically — Three extractions of copper solution with 10 ml biquinoline solution are required for complete extraction of copper. Biquinoline solution is added as 5 ml, 2.5 ml and 2.5 ml increments and shaking each time for 2 minutes.

7.3.2.1 Decompose sample in a suitable manner and dissolve in dilute hydrochloric acid. Transfer aliquot of solution containing not more

than 50 μg of copper to a separating funnel of 50 ml capacity. Add 10 ml of buffer solution. Check pH at about 6 (use narrow range pH paper or pH meter). Volume of solution should be between 15 to 20 ml. Shake with 5 ml of biquinoline solution for 2 minutes.

7.3.2.2 Allow the phases to separate and run the lower layer into another separating funnel, and extract with 2.5 ml of biquinoline solution. Take the lower layer into another separating funnel and again extract with 2.5 ml biquinoline solution. Combine the extracts, mix well, filter dry into 1 cm cell and measure absorbance at 545 nm against reagent blank with spectrophotometer. Take 0.0, 1.0, 2.0, 3.0, 4.0 and 5 ml solution [7.2.3 (b)] in 50 ml separating funnel. Add 10 ml buffer solution and proceed as above and draw calibration curve for the range 0 to 50 μg copper. Find out the copper content corresponding to the absorbance of the test solution.

NOTE — The copper complex formed with 2, 2'-biquinoline is not a strong complex. Ions that form strong complexes with copper, such as ammonium prevent the reaction of copper with 2, 2'-biquinoline and therefore should not be present during the test. If organic material is present and organic compounds are extracted by isoamyl alcohol, their yellow colour will mask the colour of the copper biquinoline complex. If samples of high organic matter are ignited at 450°C, interference from organic compounds can be prevented.

(CDC 1)